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(METALLURGICAL PROGRESS
REPORT)

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OF THE INTERIOR
BUREAU OF MINES



Albany Metallurgy Research Center
Albany, Oregon

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QUARTERLY
METALLURGY PROGRESS REPORT
Number 27

For the period of
April 1 through July 31, 1965

AEC Contract No. AT(11-1)-599

Prepared by
United States Department of the Interior
Bureau of Mines
Albany Metallurgy Research Center
Albany, Oregon

A. H. Roberson
Research Director

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SUMMARY

HIGH MELTING POINT CARBIDES (HfC)

Preliminary studies are progressing on tantalum-, columbium-, and zirconium-carbon alloys. The electrode sintering furnace has been renovated and relocated.

A total of 62 hafnium carbide castings containing about 6 to 11.5 weight-percent carbon was evaluated for integrity. The majority of these were made in shapes suitable for tensile property determination, and some of the earlier castings were made with threaded grips as an integral part of the casting. A few of these appeared to be suitable for tensile testing, but on stressing them at about 2,000° to 2,500° C, unsatisfactory results were obtained. Later, improvement in design by casting molten carbide into threaded graphite grips resulted in many castings of good quality. A limited number were stressed to fracture up to 2,180° C and the tensile strength and modulus of elasticity were determined. Two bend rupture machines were made for conducting tests at temperatures up to 2,400° C but they were little used because only a few cast specimens were produced that were considered satisfactory for this application. The emissivity of cast hafnium carbide also was determined.

THORIUM ALLOY SYSTEM

Initial work on the project consisted of a literature review of research conducted on similar problems. Two methods of producing uniform dispersions were suggested: (1) deposition of thorium on the thoria using a thermal decomposition reaction in a fluid bed, and (2) mechanically coating the oxide spheres with an industrial wax which would in turn adhere to thorium.

A fluid bed and powder blending equipment, to use with these two techniques, was constructed and existing equipment modified for use with radioactive material.

Thoria was received during the third and fourth quarters, and particle size, shape, density, and crushing strength were measured.

A group of waxes was used to coat the thoria, followed by conventional powder metallurgy methods of producing alloys with a thorium matrix. Difficulty in evaluating the uniformity of the dispersion resulted in the development of a statistical test for the degree of uniformity. Thus far, only random dispersions have been obtained.

COLD-MOLD INDUCTION MELTING

Successful development of equipment and techniques for induction melting of reactive metals in a cold copper crucible has resulted in submission of a patent application. Efforts are now underway to increase the scale of operations.

INVESTIGATION OF THE SYSTEM UC-UN-UO

Equipment and techniques have been further refined in an attempt to improve composition control of the uranium oxycarbides under study. The effect of stoichiometric unbalance of oxycarbides on such properties as lattice parameter, decomposition pressure, density and phase relationships is currently being examined.

TUNGSTEN-RHENIUM TUBING DEVELOPMENT

Bureau of Mines' facilities were used for the preparation of tungsten-rhenium castings and ingots as blanks for extrusion. Although the Bureau's assigned tasks were successfully completed, meaningful progress toward ultimate objectives was frustrated because (1) change of the goals before they could be attained, and (2) responsibility for some of the final stages of work and the reporting of results were assigned to groups outside of the Bureau.

The primary objective of this project is to prepare by powder metallurgy, tungsten-25 rhenium alloy sleeves which can be subsequently extruded and drawn into engineering quality tubing. An added objective is to evaluate prealloyed tungsten-25 rhenium powders.

RHENIUM AND RHENIUM-BASE ALLOYS

Attempts to extrude both the powder metallurgy and the arc melted W-25 Re billets were unsuccessful with the extrusion parameters selected. Other attempts may be possible after reconditioning of the billets.

end

QUARTERLY
METALLURGICAL PROGRESS REPORT

Number 27

AEC Contract No. AT(11-1)-599
Activity Number 4420

HIGH MELTING POINT CARBIDES (HfC)

R. A. Beall and H. Kato, Project Coordinators

Casting - R. P. Adams

The objectives of this project are (1) to improve the equipment and technique for melting and casting high-melting-point metal-carbon alloys, and (2) to spin cast suitable shapes in these alloys for the evaluation of their mechanical and thermal properties at elevated temperatures.

These objectives have been met to the extent that the equipment and technique have been improved and spin castings have been made in the desired shapes through the use of a new mold design. However, additional improvement is to be desired in the integrity of the castings. It is hoped that with an enlarged and redesigned furnace, using the experience gained from the present modified version, it will be possible to consistently pour better quality castings during the next fiscal year.

In the first quarter the furnace was modified and scaled up to accommodate a larger crucible in anticipation of the various shapes desired. Molds were designed and fabricated for castings of various dimensions including tensile specimens. As larger castings were made, the various furnace components were of necessity improved.

During the second quarter, a tensile specimen design was selected with either ball or threaded ends for use in the Marquardt testing machine. A novel mold design was developed for this specimen which made it possible to cast pieces with enlarged ends and a reduced section in the center without breaking from shrinkage. The essential component of

this new type mold was the segmented center section, held together with iron wire which melts at the proper time. The resultant collapse permits lateral shrinkage after solidification.

This mold was further improved in the third quarter to yield a reduced section one inch long and with externally threaded graphite mold ends that are left on the castings. These sections were screwed into the grips on the testing machine and furnished thermal insulation between the hot specimens and the grips. The one inch long reduced section in the casting made it possible to measure elongation. A series of hafnium carbide specimens of the improved design for elevated temperature evaluation was completed.

A series of metal-carbon alloys, including tantalum, columbium, and zirconium, as well as hafnium is now being studied. It was found that the first tantalum-carbon and columbium-carbon electrodes broke up badly in the furnace from thermal shock. Apparently this was due to the use of metal powder instead of sponge which is normally used in the preparation of hafnium-carbon electrodes. Various combinations of metal turnings, crushed carbide, and metal powder along with fine graphite have been pressed into small compacts for evaluation. Considerable progress has been made in developing tantalum-carbon and columbium-carbon electrodes which are stable under melting conditions.

Electrodes of graphite and electrodes of tantalum metal were tried with little success. With graphite electrodes, the furnace was filled with a luminous smoke cloud or "ball of fire," making it almost impossible to observe the melting operations. Both types of electrodes changed the composition of the pool to such an extent that they were deemed impractical.

The high-temperature, high-vacuum resistance furnace, which is used to sinter metal-carbon electrodes, has been completely renovated and relocated.

Quality Evaluation of Carbide Specimens - M. I. Copeland

A total of 27 hafnium carbide castings, made for tensile determinations, were evaluated for quality; 11 of these were considered satisfactory for test purposes, 8 were doubtful, and 9 unsatisfactory. None of the castings were tested in the Marquardt tensile machine this quarter as it was unavailable because of the priority of other tests.

Three columbium carbide and two tantalum carbide castings made for tensile testing were evaluated. All but one casting, made of columbium carbide, was unsatisfactory because of cracks and/or porosity. These castings were made primarily for determining melting and casting parameters.

Studies to determine the minimum melting point of hafnium carbide with selected refractory metals were completed. Solidus temperatures of 1,655°, 1,760°, 1,790°, 2,310°, 2,350°, 2,680°, and 2,730° C were noted when hafnium carbide, containing 9.6 weight-percent total carbon, was placed in sight holes in titanium, zirconium, vanadium, columbium, molybdenum, tantalum, and tungsten, respectively, and heated.

A study of the reactivity of tantalum carbides with the air was started. The temperature at which tantalum carbide was found to noticeably oxidize occurred between 500° and 550° C. Rates of oxidation at higher temperatures were being established.

The design of a furnace and accessory equipment for tensile testing carbide castings was started, as was also a unit for measuring elongation at temperatures up to 2,500° C or higher. Construction of these units would be expected during the next fiscal year.

MANUSCRIPTS

1. Iron-Chromium-Gadolinium Alloys, by M. I. Copeland, W. Barstow, and H. Kato. This paper is being reviewed locally.
2. Hafnium-Iridium Alloy System, by D. Goodrich, D. Deardorff, M. Copeland, and H. Kato. The manuscript is in preparation.
3. Investigation of Hafnium Carbon System, by D. Deardorff, M. Copeland, and H. Kato. This paper has been reviewed locally.

THORIUM ALLOY SYSTEMS

H. Kato, Project Coordinator
S. A. O'Hare

The development of methods of uniformly dispersing 15 to 50 volume percent thorium in a thorium matrix, the determination of fabrication schedules, and a study of the properties of the alloys are the goals of this project.

Thoria was received during the third and fourth quarters, and particle size, shape, density, and crushing strength were measured.

The final lot of thorium was obtained from Oak Ridge National Laboratory during the last quarter, and has an approximate particle size of 1.6 microns.

A fluid bed technique is being adapted to coat this final lot of thorium. The method is based on the formation of thorium tetraiodide gas at 450° C, and the thermal decomposition of this gas at 900° C. It is hoped to accomplish this thermal decomposition in a fluid bed of thorium. The equipment has been assembled, and two runs attempted. However, both trials were stopped because of leakage of iodine gas. Some coating appears to be occurring based on the surface appearance of the thorium.

A measure of the crushing strength of the oxide was conducted to prevent future cracking of spheres during compacting. A 500-pound load cell was incorporated on a Rockwell hardness testing machine, and the crushing load for the spheres was measured. Values obtained ranged from 10 pounds to 21 pounds, depending on the particle size.

Wax coatings of the microspheres have continued also during this final quarter. The major difficulty is preventing the spheres from adhering to each other. No compacts with uniform dispersions have yet been attained.

MANUSCRIPTS

No manuscripts scheduled.

COLD-MOLD INDUCTION MELTING

R. A. Beall, Project Coordinator
P. G. Clites

The objective of this project is to develop a furnace for induction melting reactive metals in a water-cooled copper crucible. During the first 2 quarters of this fiscal year, tests were completed on melting units designed to produce 3/4-inch and 1-inch-diameter ingots. Relatively sound ingots of titanium and zirconium were produced in these units, and on the basis of the results obtained, a patent application was submitted through the U. S. Atomic Energy Commission San Francisco Operations Office for the melting equipment developed. During the second half of the year this equipment has been enlarged to accommodate 1-1/2-inch-diameter ingots, and a larger power supply was obtained and installed. Tests are now being conducted in this equipment and will continue during the first part of Fiscal Year 1966.

The results of tests completed with the 1-1/2-inch unit have been only moderately successful thus far. Early tests with the new power supply were frequently interrupted by high voltage discharges within the furnace. This difficulty has been eliminated by improvement of the insulation used between the high voltage leads to the work coil and the furnace shell and by eliminating a number of small leaks in the furnace. It is now possible to operate the unit for sustained periods with no difficulty.

Tests are now in process in which minor alterations of the melting unit are being made in an attempt to improve the efficiency of the unit. During tests conducted, heating of the upper surface of titanium and zirconium ingots has been insufficient to maintain a full, deep molten pool unless the ingot was raised considerably above the top of the water-cooled copper crucible. With the ingot in a raised position, a good pool forms but the ingot has a tendency to mushroom which makes withdrawal difficult. However, during several tests it was observed that a significant levitating action, similar to that noted with the smaller units, occurred when the ingot was in a certain position. This levitating action forces the center of the pool upward and eliminates the mushrooming effect. Minor alterations to the melting unit are now being made in an effort to increase this levitating action, thereby allowing a full pool of molten metal to be maintained.

These alterations to the unit will be continued during the first quarter of next year.

In addition to the work conducted in this area, the manuscript, "A Study of Heat Transfer to Water-Cooled Copper Crucibles During Vacuum Arc Melting" was completed and is now undergoing local review. This manuscript will be published as a U. S. Bureau of Mines Report of Investigations.

INVESTIGATION OF THE SYSTEM UC-UN-UO

Hal J. Kelly, Project Coordinator

Jack L. Henry, Danton L. Paulson and Robert Blickensderfer

The objective of this program is the investigation of the pseudo-ternary system UC-UN-UO in connection with its usefulness in the production of high temperature nuclear reactor fuels. Of particular interest are the subliquidous phase relationships within the system and the evaluation of such properties as thermal stability, thermal conductivity, uranium density and hot-hardness.

Considerable time has been devoted this quarter to the study of sintering techniques and to the examination of the effects of variation in stoichiometry with respect to carbon and uranium at various oxygen levels in the uranium oxycarbides.

Materials

Investigation has continued on the preparation of fine uranium powder by single and by multiple hydriding and dehydriding of pickled uranium turnings. Particle size analyses indicate that little is gained by employing a third hydriding-dehydriding cycle. Multiple hydriding and dehydriding produces a finer and more sinterable powder, but results in a higher level of oxidation. Oxygen content ranges from about 1,000 to 5,000 ppm depending upon the number of cycles and the quality of the glove box atmosphere during powder handling.

Equipment and Facilities

An argon circulation system has been installed on the glove box to increase the efficiency in removal of oxygen and moisture by the getter-furnace and molecular sieve. An extension has also been added to the vacuum antechamber to provide for the introduction and removal of larger equipment such as the hydriding assembly. An oxygen indicating apparatus has been constructed according to a publication by P. S. Davis (1). The method is only semiquantitative, but is adequate to indicate when the oxygen content of the glove box atmosphere is greater or less than 100 ppm. Pyrophoric materials are handled only when the atmosphere contains less than 100 ppm oxygen.

The outgassing rate of the vacuum sintering furnace has been substantially reduced by modifying the water cooling system for the power connection to the heater element. A small segment of plastic tubing carrying

coolant water between the electrical terminals was originally located within the vacuum chamber. By use of two additional vacuum feed-throughs the plastic tube has been relocated outside the chamber. Pressure build-up at $1,700^{\circ}\text{C}$ was thus reduced from about 3 microns per minute to about 0.5 microns per minute. Rates given are under sintering conditions in which no initial high temperature outgassing is carried out.

The seamless molybdenum tube decomposition pressure measurement apparatus described previously has been installed and tested. The outgassing rate of the attachment is about 0.07 micron liter per minute at $1,700^{\circ}\text{C}$, much less than that of the sintering furnace chamber, but because of its smaller volume the pressure build-up is still 3 to 4 microns per minute. The background pressure inherent in the sintering furnace made measurements of decomposition pressure below 50 or 100 microns unreliable by the direct method.

An electric clock motor coupled to a step-function speed reductor has been installed on the control of the saturable reactor transformer used to power the vacuum sintering furnace. As a result, the heat-up rate during specimen sintering is much more uniform.

System Study

Fifty-six oxycarbide specimens have been studied during this quarter in an attempt to gain more information on sintering variables and on the effect of slight unbalance of stoichiometry.

Until recently, this study was concerned only with stoichiometric compositions in which the number of atoms of uranium is equal to the sums of the atoms of carbon and oxygen or to the sums of the atoms of carbon, oxygen and nitrogen. It has become apparent however that many compositions which were synthesized to be stoichiometric have turned out to be nonstoichiometric. This difference from the intended composition is due primarily to oxidation of uranium powder during body synthesis and to a small degree of decomposition and uranium vaporization during sintering. A good deal of scatter has been noted in the lattice parameters even among specimens having the same intended composition. This same difficulty in controlling composition may be responsible for many discrepancies in data found in the published literature.

Twenty-five tests were run in which bodies of nominal composition $\text{UC}_{.70}\text{O}_{.30}$ were fired under various conditions of time, temperature and pressure and in which some of the bodies were refired. Discrepancies seem to occur with specimens which have been fired by raising the temperature to $1,700^\circ\text{C}$ without an appreciable hold at a lower temperature. Therefore, several bodies were prefired to examine the effect of temperatures in the range of 800° to $1,200^\circ\text{C}$. Distinct exothermic reactions were observed in this set of experiments. The first exothermic reaction occurred near 800°C while the second and stronger reaction occurred near the melting point of uranium, $1,135^\circ\text{C}$. The first reaction appears to be between uranium and graphite to form the monocarbide. The lattice parameters of compositions fired near 800°C were 4.959 to 4.960 \AA . The reduction of free UO_2 content seems to indicate that the second exothermic reaction may be due to the reaction of the monocarbide with the excess UO_2 and U to take oxygen into the structure. This is further evidenced by a decrease in lattice parameter upon firing to $1,135^\circ$ to $1,170^\circ\text{C}$.

The X-ray diffraction lines of these prefired bodies are rather broad which might indicate incomplete reaction and poor crystal formation.

Thirteen bodies were refired at different temperatures after initial prefiring. Weight losses were low, indicating insignificant decomposition. Refiring at $1,135^\circ$ to $1,170^\circ\text{C}$ reduced the lattice parameter and free UO_2 somewhat. Refiring at $1,350^\circ\text{C}$ reduced the parameter and UO_2 content still more. Refiring near $1,700^\circ\text{C}$ for 1 and 2 hours lowered the lattice parameter to about 4.953 \AA and resulted in reaction of nearly all the free UO_2 . Two of the $\text{UC}_{.70}\text{O}_{.30}$ bodies were essentially single phase.

It would appear from these firings that the lattice parameter for the composition $\text{UC}_{.70}\text{O}_{.30}$ is near 4.953 \AA . However, some parameters have been lower in former bodies. It is possible that they could be nonstoichiometric. Several older bodies having abnormally low parameters were annealed at $1,000^\circ\text{C}$ for 2 hours in vacuum in an attempt to precipitate any excess uranium in the oxycarbide phase. Essentially no change was noted in free UO_2 content or lattice parameter. The annealing temperature may have been too low or the time too short.

Because of our interest in the possible effects of stoichiometric unbalance of the oxycarbides on such properties as lattice parameter, decomposition pressure, density and phase relationships, a new series of experiments was started. Several compositional traverses representing variations in stoichiometry from hyper to hypostoichiometric at various oxygen levels have been made. Thus far, traverses at oxygen levels of 5, 10, 12.5 and 15 mole percent have been examined. These data are presented in table 1. All of the specimens were sintered individually in the same manner. The temperature was slowly raised to approximately $1,200^{\circ}\text{C}$ in a vacuum of 10^{-5} to 10^{-6} torr and held for 1 hour. The system then was isolated from the diffusion pump and the temperature increased to $1,700^{\circ}\text{C}$ where it was held for 2 hours. Pressure in the furnace chamber was allowed to build up from the gas produced by the normal decomposition of the body. The specimen was then furnace-cooled to room temperature. The specimen cooled from $1,700^{\circ}$ to 800°C in three minutes. From previous experiments, this sintering schedule appears to be the most satisfactory in preventing uranium loss to the container crucible and in minimizing decomposition of the oxycarbide. Although this study is incomplete the following observations are worthy of mention:

1. As the composition changes from hyperstoichiometric (carbon-rich) to hypostoichiometric (uranium-rich) at all oxygen levels, the sintered density increases, the decomposition pressure decreases and the lattice parameter decreases.
2. The decomposition pressure decreases as the oxygen content of slightly hyperstoichiometric compositions is increased.
3. Although the single phase range appears to extend well into the hyperstoichiometric region at the 5 mole percent oxygen level, etching reveals a Widmanstätten pattern of UC_2 . The UC_2 is apparent in all hyperstoichiometric bodies at this oxygen level, increasing in amount with increased carbon content.
4. The single phase range does not extend far into the hyperstoichiometric region at higher oxygen levels nor into the hypostoichiometric region at any oxygen level thus far examined.
5. Although the 15 mole percent oxygen level should be near the limit of oxygen solubility, no three phase bodies were found on the traverse. Bodies were two phase, oxycarbide and UO_2 and oxycarbide and U.

TABLE 1. - Composition traverse study

| Body no. | Nominal composition | | Lattice parameter Å | density g/cm ³ | Percent free UO ₂ (X-ray) | Other phases found in UCO microstructure | Pressure developed during sintering, microns |
|----------|---------------------|------|---------------------|---------------------------|--------------------------------------|--|--|
| | U | C | O | | | | |
| 160 | .45 | .50 | .05 | 4.9610 | 9.39 | 0.2 | Widmanstätten UC ₂ |
| 161 | .46 | .49 | .05 | 4.9610 | 9.69 | 0.2 | do |
| 153 | .465 | .485 | .05 | 4.9607 | 9.74 | 0.2 | do |
| 162 | .465 | .485 | .05 | 4.9602 | 9.39 | 0.4 | Trace Widmanstätten UC ₂ |
| 154 | .47 | .48 | .05 | 4.9608 | 9.70 | 0.2 | do |
| 155 | .475 | .475 | .05 | 4.9606 | 9.97 | 0.2 | do |
| 143 | .48 | .47 | .05 | 4.9596 | 9.45 | 0.2 | do |
| 144 | .49 | .46 | .05 | 4.9590 | 9.94 | 0.3 | do |
| 145 | .50 | .45 | .05 | 4.9573 | 10.2 | 0.3 | none |
| 158 | .50 | .45 | .05 | 4.9572 | 11.0 | 0.2 | Trace free U |
| 156 | .505 | .445 | .05 | 4.9558 | 10.9 | 0.3 | do |
| 157 | .51 | .44 | .05 | 4.9566 | 11.4 | 0.3 | Free U |
| 146 | .52 | .43 | .05 | 4.9580 | 11.7 | 0.3 | do |
| 147 | .54 | .41 | .05 | 4.9535 | -- | 0.3 | do |
| 148 | .48 | .42 | .10 | 4.9594 | 9.46 | 0.7 | Free UO ₂ |
| 149 | .49 | .41 | .10 | 4.9583 | 9.44 | 0.6 | do |
| 150 | .50 | .40 | .10 | 4.9560 | 9.67 | 0.3 | none |
| 151 | .505 | .395 | .10 | 4.9559 | 9.84 | 0.1 | Free U |
| 152 | .51 | .39 | .10 | 4.9553 | 10.3 | 0.2 | do |
| 163 | .49 | .385 | .125 | 4.9557 | 9.23 | 0.3 | none |
| 165 | .50 | .375 | .125 | 4.9551 | 10.3 | 0.2 | Free U |
| 166 | .505 | .37 | .125 | 4.9545 | 11.4 | 0.1 | do |
| 167 | .49 | .36 | .15 | 4.9538 | 9.20 | 0.6 | Free UO ₂ |
| 168 | .495 | .355 | .15 | 4.9519 | 9.59 | 0.2 | Trace free U |
| 169 | .50 | .35 | .15 | 4.9525 | 10.1 | 0.3 | Free U |
| 170 | .505 | .345 | .15 | 4.9524 | 10.7 | 0.3 | do |

A limited amount of work has been done on the production of high density uranium oxycarbide bodies by hot pressing. Both molybdenum and tantalum lined graphite dies have been employed, but reaction between the specimen and the die assembly has occurred in both cases. A tungsten die is being constructed for further experiments.

References

1. - Davis, P. S. "Detection of Traces of Oxygen in Gases, The Methylene Blue Method," Metallurgia, 62, 369, 1960, pp. 49-50.

TUNGSTEN-RHENIUM TUBING DEVELOPMENT

H. Kato and R. A. Beall, Project Coordinators

Melting - E. D. Calvert

The purpose of this program is to develop a technology for producing sound, arc-cast ingots of W-25 Re alloy suitable for extrusion and ultimate conversion to good quality alloy tubing.

The goal of this work, begun in Fiscal Year 1964, was to develop methods for producing hollow, W-25 Re alloy tube blanks by skull casting for direct fabrication to tubing, and as an alternate method, to prepare arc-cast alloy ingots which could be machined to form hollow billets for tube extrusion and secondary fabrication. Equipment was designed and assembled during Fiscal Year 1964, and several small-scale hollow tube blanks were cast. Three of these blanks were extruded and evaluated at Nuclear Metals Corporation and were found to be inferior to similar material prepared by powder metallurgy techniques because of excessively high carbon content and the presence of sigma phase. Before quality of the skull-cast products could be up-graded, interest in skull casting waned, and research was directed to preparation of arc-cast ingots for conversion to tubing for evaluation.

Insofar as effective melting parameters had been established during the skull-casting phase of the program, preparation of ingots by arc melting was fairly straightforward. Of prime interest was the control of variables within the melting process to effect changes in grain morphology, soundness, and homogeneity.

Three sets of 3-inch-diameter ingots were cast in duplicate (6 total). Major variations were melt rate, magnetic stirring, and cooling rate. The obvious effects of melting practices were seen in the as-cast ingots. Those produced at the highest power were superior in ingot side-wall condition and yield to finished product. Better homogeneity appeared to result from the faster rates of stirring. Whether or not grain size, mechanical properties, or ease of fabrication were effected by operating variables is unknown. It is doubtful if these latter effects will ever be evaluated as the ingots are being fabricated by an outside cooperating AEC agency and the major interest is in development of extrusion techniques and evaluation of the wrought product.

Four of the six ingots melted were conditioned for primary extrusion. Two were prepared for tube extrusion and two for extrusion to sheet bar. The finished billets were transmitted to Oak Ridge National Laboratory where work is proceeding. To date no satisfactory results have been obtained because of difficulties in establishing extrusion parameters (i. e. temperature, extrusion speed, tooling, reduction ratio, and lubricant) rather than because of billet quality. No attempts have yet been made to extrude the tube shell blanks. They are to be coated with pyrolytic tungsten in order to obtain tungsten oxide for lubrication. Tungsten oxide has been a successful lubricant for the extrusion of unalloyed tungsten. Disposition of the six ingots prepared during Fiscal Year 1965 is presented in table 2.

TABLE 2 - Disposition of arc-melted W-25 Re ingots prepared in F. Y. 1965

| <u>Heat number</u> | <u>Machined dimensions</u> | | | <u>Disposition</u> |
|--------------------|----------------------------|-----------------------|--------------------|--|
| | <u>O. D. , inches</u> | <u>Height, inches</u> | <u>Weight, kgs</u> | |
| 24295 | 2-3/4 | 3-7/8 | 7.4 | At Oak Ridge; attempts to extrude bare ingots at 3,990° F (2,200° C) at 10:1 stalled press. Re-extrusions planned. |
| 24302 | 2-27/32 | 5-7/32 | 10.4 | |
| 24303 | 2-5/8 | 5-5/8 | 9.7 | On hand at A. M. R. C. |
| 24304 | 2-25/32 | 5-19/32 | 10.6 | |
| 24330 | <u>1/</u> | <u>1/</u> | <u>1/</u> | Eloxed by G. E. (N. M. P. O.) to form sleeves 7/8-inch I. D. ; at Oak Ridge to be extruded. |
| 24332 | <u>1/</u> | <u>1/</u> | <u>1/</u> | |

1/ Indicates dimensions are not available.

Powder Metallurgy - G. Asai

The primary objective of this project is to prepare by powder metallurgy, tungsten-25 rhenium alloy sleeves which can be subsequently extruded and drawn into engineering quality tubing. An added objective is to evaluate prealloyed tungsten-25 rhenium powders.

Using the powder metallurgy procedure which was developed in FY 1964 for the preparation of tungsten-25 rhenium alloy sleeves, 56 sleeve extrusion blanks were prepared. Thirty-three sleeves were shipped to Nuclear Metals, Inc. for further fabrication and evaluation. Prealloyed tungsten-25 rhenium powders which had been prepared by Curtiss-Wright and by Union Carbide at Oak Ridge, Tennessee were evaluated. In general, the three lots of prealloyed powders contained 5 to 30 percent of second phase by volume, but of significance was that: (1) individual particles were actually alloyed to various degrees, and (2) specimens free of second phase could be obtained when sintering temperatures as low or lower than that required for elemental mixtures were used. The prealloyed powders were processed without difficulty to yield sintered sleeves of a quality comparable to those currently being made from blended mixtures of elemental tungsten and rhenium.

Sintered Sleeves for Nuclear Metals, Inc. (NMI)

During FY 1964 relatively short tungsten-25 rhenium alloy sleeves were prepared by powder metallurgy, during which the effects of particle size, blending procedures, and sintering conditions were investigated. The results of this investigation were summarized in the report for the quarter ending September 1964. Using the method developed in FY 1964, 56 tungsten-25 rhenium alloy sleeve extrusion blanks were prepared during the past year. Thirty-three of the sleeves were shipped to NMI and the remainder are being kept on hand pending further investigations. Fifty-two sleeves had a nominal size of 2.885 cm OD by 0.254 cm wall by 20.3 cm long. Four sleeve extrusion blanks of this size are shown in figure 1. The other four had a nominal size of 3.81 cm OD by 0.4 cm wall by 20.3 cm long. The procedure used consisted of four cycles of tumble and screen blending 4.2 micron tungsten powder with 2.2 micron rhenium powder, isostatic pressing the vibration-loaded mixture at 2,110 kg/cm², and sintering the sleeves by heating for 15 hours at 2,400° C in hydrogen. Metallography, density, and rhenium content data obtained on single specimens from each sleeve showed that:

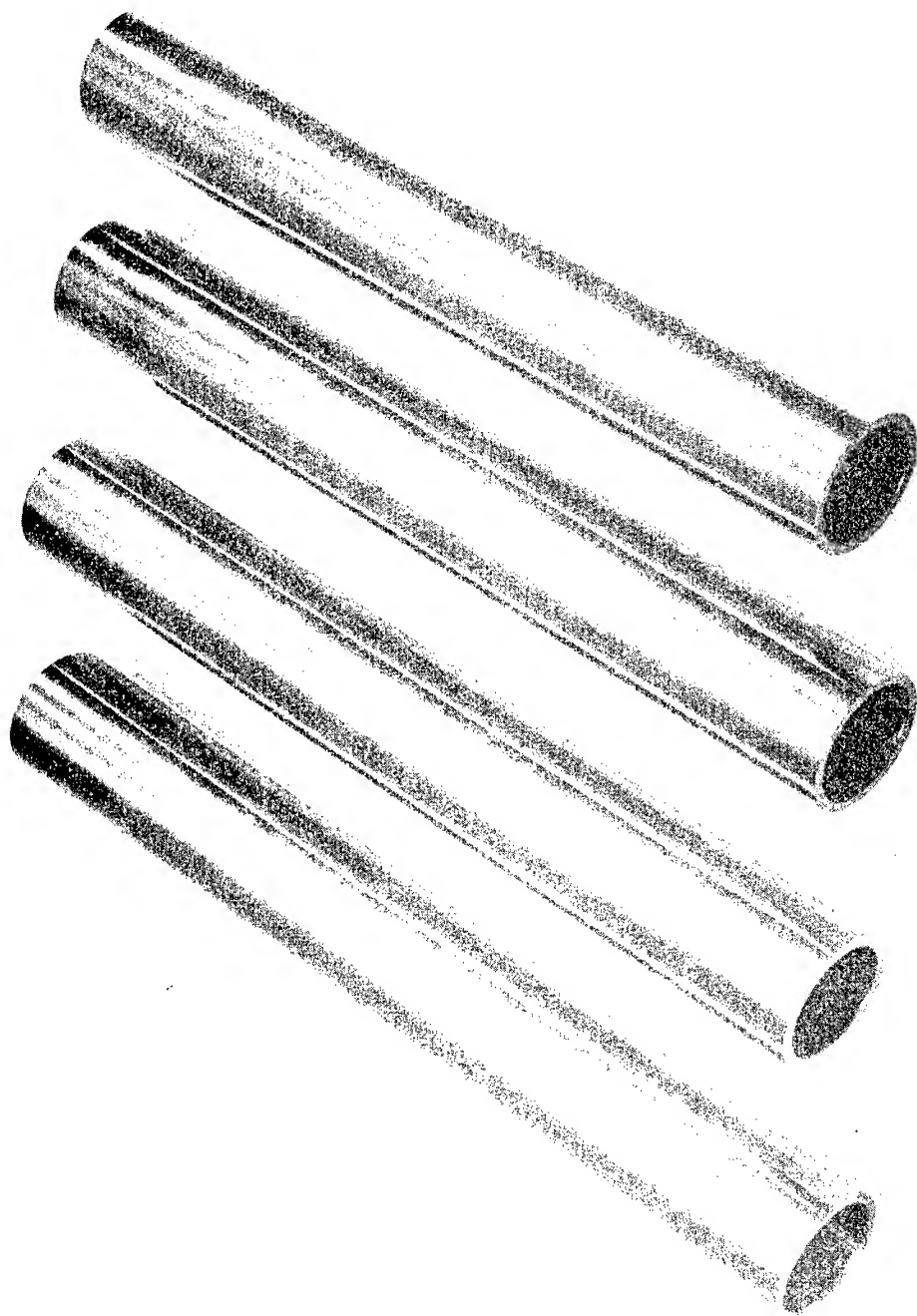


FIGURE 1. - Finish machined tungsten-25 rhenium sleeve extrusion blank.
(2.885 cm OD by 0.254 cm Wall by 20.3 cm)

1. The sleeves were free of second phase and that the grain size ranged from ASTM No. 5 to 8, as shown in figure 2.
2. The densities ranged from 18.8 to 19.4 g/cc with an average of 19.15 g/cc which corresponds to a value of 97.5 percent of theoretical density.
3. The rhenium content ranged from 24.1 to 25.4 with an average of 24.97 percent.

Evaluation of Prealloyed Powders

Prealloyed tungsten-25 rhenium powders prepared by Curtiss-Wright and Union Carbide (ORGDP) were evaluated primarily to determine their suitability for use in the preparation of sintered sleeves. The as-received powders were examined by Fisher subsieve, turbidimetric, Brunauer-Emmett-Teller (B. E. T.), X-ray diffraction, electron microprobe, chemical, and spectrographic analyses. The sinterability of these materials was studied by sintering sections from sleeves isostatically pressed at 2,110 kg/cm². Sintering tests were made at 1,500°, 1,800°, 2,000°, 2,200°, and 2,400° C in hydrogen. The specific surface area, average particle size and particle size distribution for the as-received prealloyed powders are shown in table 3 with corresponding data for W-25 Re powders made by blending elemental tungsten and rhenium. X-ray diffraction and microprobe analyses of the as-received powders are summarized in table 4. Specimens from sections sintered at various temperatures were evaluated for density by the Archimedes method; for rhenium content by X-ray fluorescent analysis; grain size by metallography; and presence of second phase by metallography, X-ray diffraction, and electron microprobe analyses. These results are summarized in table 5. Photomicrographs of prealloyed specimens sintered in hydrogen at 1,800°, 2,000°, 2,200°, and 2,400° C are shown in figures 3, 4, and 5, which can be compared with the photomicrographs in figure 6 of sintered sections made from 1-1/4-micron tungsten blended with 2.2-micron rhenium.

Although there were differences in particle size in molybdenum, nickel, iron, and rhenium contents among the three lots of prealloyed powders, the following similarities were noted. The three lots of prealloyed powders contained 5 to 30 percent of second phase by volume but of significance was that: (1) the majority of individual particles were actually alloyed to various

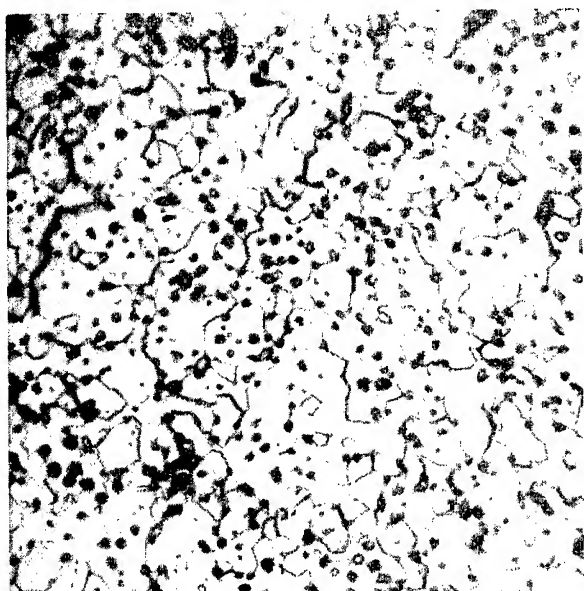


Sample No. B-916 Magnification: 500 X

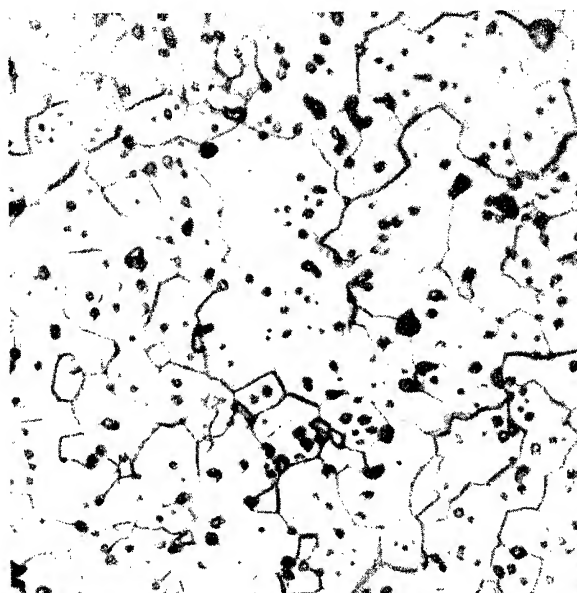
Identification: T-177 Etch: Murakamis

Remarks: As-sintered; dark spots are voids;
ASTM grain size No. 6; 97.6 percent of theoretical
density.

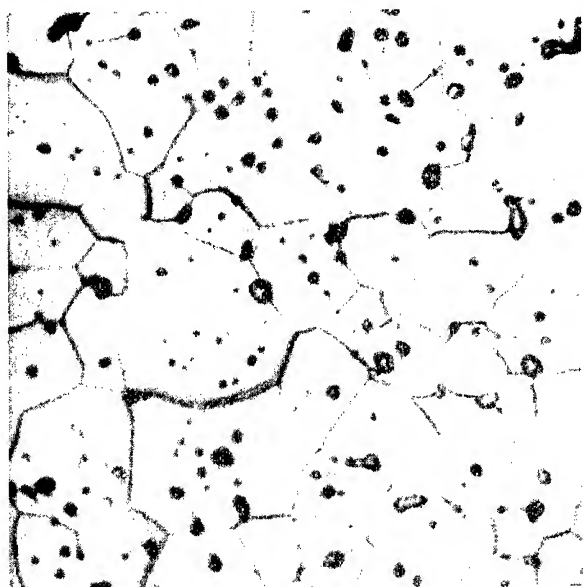
FIGURE 2. - Photomicrograph of Typical Tungsten-25 Rhenium Sleeve (T-177)
Sintered 15 Hours at 2,400° C in Hydrogen



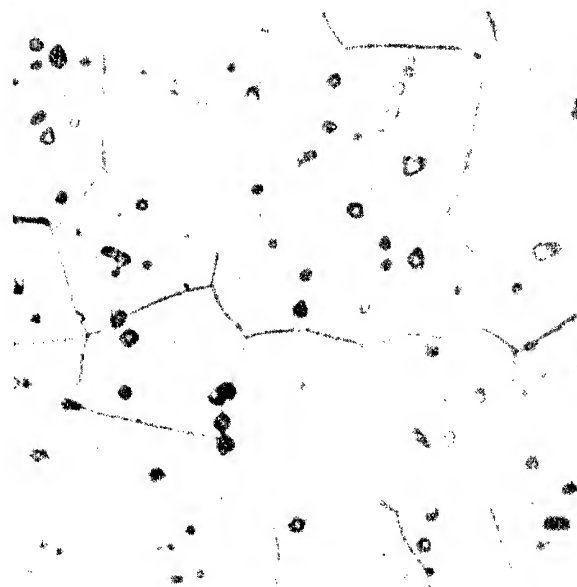
Sample No. B-927 Mag: 500 X
Ident. T-150-18 Etch: Murakamis
Remarks: As-sintered 4 hrs at 1,800° C.
 Dark spots are voids; ASTM grain size
 No. 10 to 11; 89-pct of theoretical density.



Sample No. B-928 Mag: 500 X
Ident. T-150-20 Etch: Murakamis
Remarks: As-sintered 4 hrs at 2,000° C.
 Dark spots are voids; ASTM grain size
 No. 9; 92.6-pct of theoretical density.

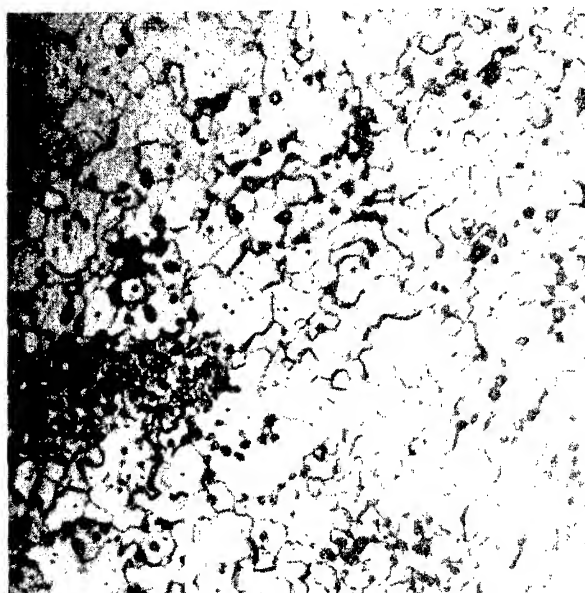


Sample No. B-929 Mag: 500 X
Ident. T-150-22 Etch: Murakamis
Remarks: As-sintered 4 hrs at 2,200° C.
 Dark spots are voids; ASTM grain size
 No. 8; 94.6-pct of theoretical density.



Sample No. B-590 Mag: 500 X
Ident. CW-N Etch: Murakamis
Remarks: As-sintered 4 hrs at 2,400° C.
 Dark spots are voids; ASTM grain size
 No. 5-6; 95.4-pct of theoretical density.

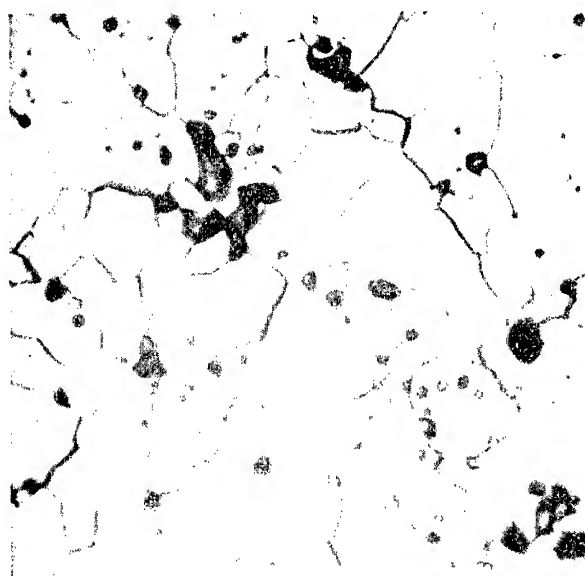
FIGURE 3. - Photomicrographs of Curtiss-Wright Prealloyed Tungsten-25 Rhenium
 Sintered at Various Temperatures in Hydrogen.



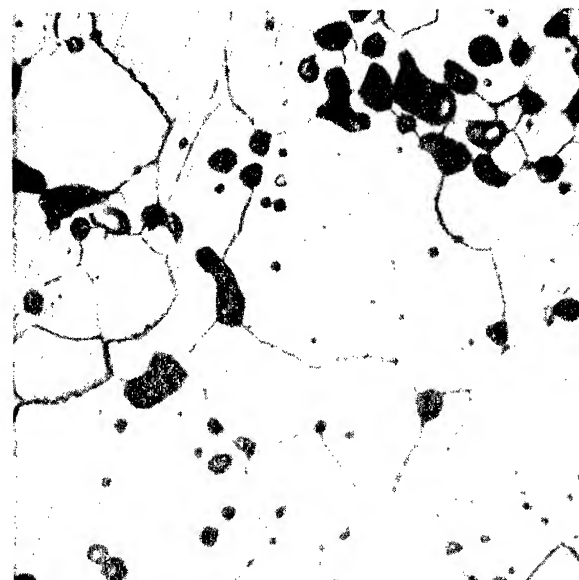
Sample No. B-936 Mag.: 500 X
Ident. T-152-18 Etch: Murakamis
Remarks: As-sintered 4 hrs at 1,800° C.
 Two high-Re areas shown; ASTM grain
 size No. 11; 93-pct of theoretical density.



Sample No. B-937 Mag.: 500 X
Ident. T-152-20 Etch: Murakamis
Remarks: As-sintered 4 hrs at 2,000° C.
 Dark spots are voids; ASTM grain size
 No. 10; 94-pct of theoretical density.

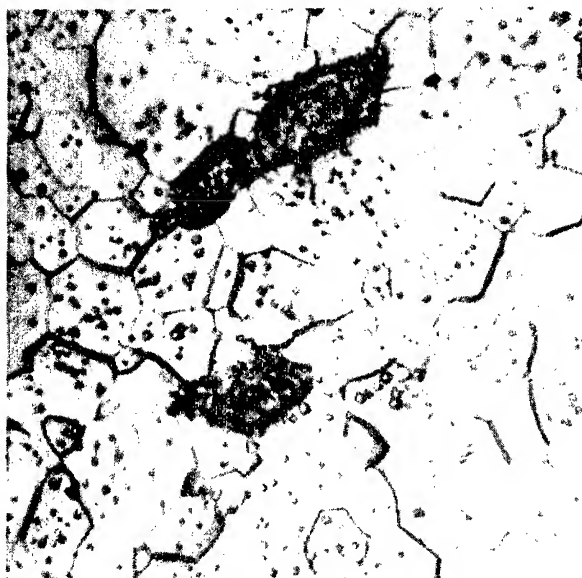


Sample No. B-938 Mag.: 500 X
Ident. T-152-22 Etch: Murakamis
Remarks: As-sintered 4 hrs at 2,200° C,
 Dark spots are voids; ASTM grain size
 No. 9; 90-pct of theoretical density.



Sample No. B-939 Mag.: 500 X
Ident. T-152-N Etch: Murakamis
Remarks: As-sintered 4 hrs at 2,400° C.
 Dark spots are voids; ASTM grain size
 No. 5-9; 93-pct of theoretical density.

FIGURE 4. - Photomicrographs of Union Carbide (ORPA-1) Prealloyed Tungsten-
 25 Rhenium Sintered at Various Temperatures in Hydrogen



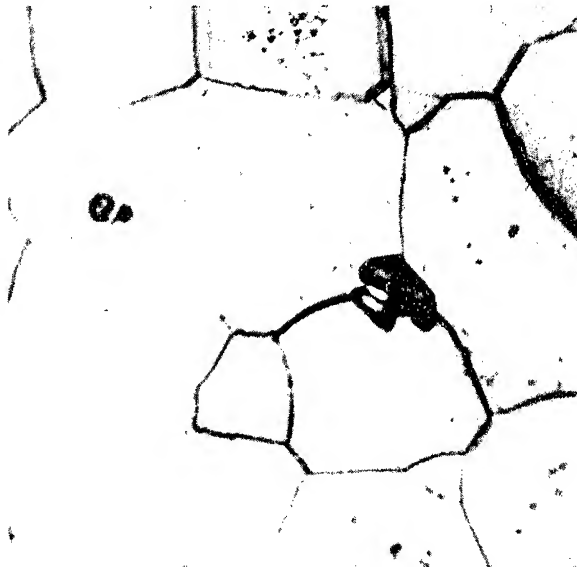
Sample No. B-951 Mag.: 500 X
Ident. T-180-18 Etch: Murakamis
Remarks: As-sintered 4 hrs at 1,800° C.
 Dark spots are voids; two high void areas
 contain 50-pct Re; ASTM grain size No. 9;
 97.1-pct of theoretical density.



Sample No. B-952 Mag.: 500 X
Ident. T-180-20 Etch: Murakamis
Remarks: As-sintered 4 hrs at 2,000° C.
 High porosity area is high in Re; ASTM
 grain size No. 8; 97.5-pct of theoretical
 density.

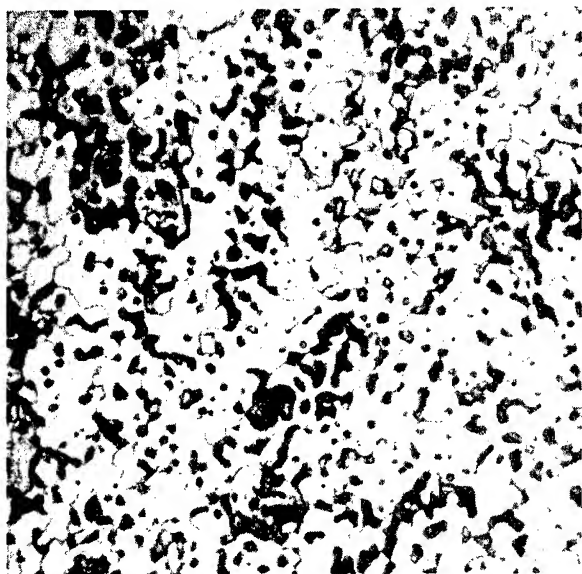


Sample No. B-953 Mag.: 500 X
Ident. T-180-22 Etch: Murakamis
Remarks: As-sintered 4 hrs at 2,200° C.
 High porosity area contains 34-pct Re;
 ASTM grain size No. 5-6; 98.6-pct of
 theoretical density.

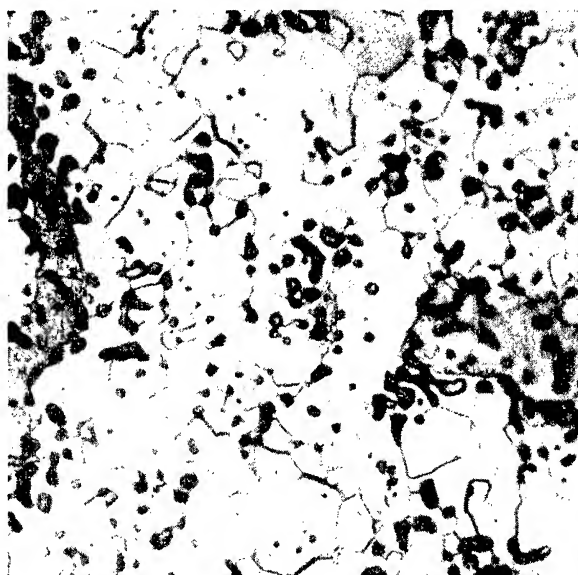


Sample No. B-954 Mag.: 500 X
Ident. T-180-N Etch: Murakamis
Remarks: As-sintered 4 hrs at 2,400° C;
 ASTM grain size No. 5-6; 99.5-pct of
 theoretical density.

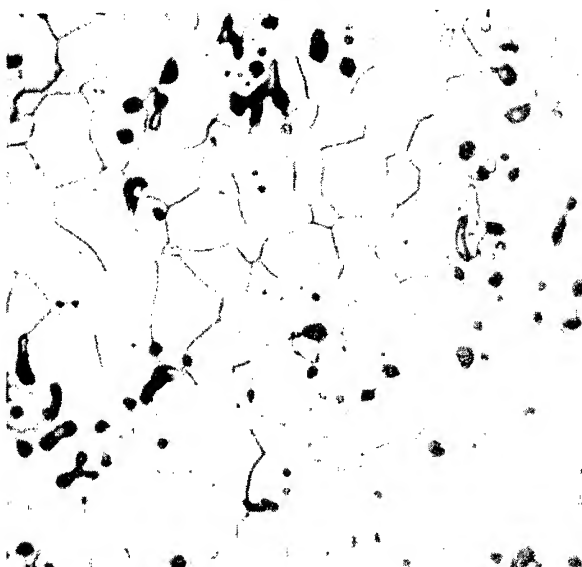
FIGURE 5. - Photomicrographs of Union Carbide (ORPA-2) Prealloyed Tungsten-25 Rhenium Sintered at Various Temperatures in Hydrogen



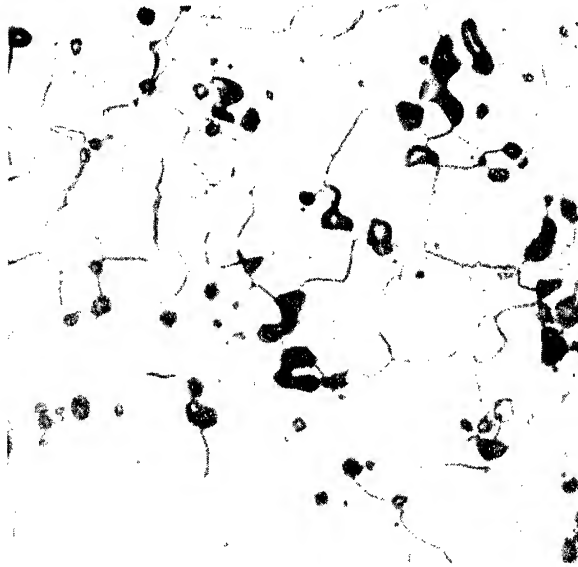
Sample No. B-941 Mag.: 500 X
 Ident. T-178-18 Etch: Murakamis
 Remarks: As sintered 4 hrs at 1,800° C.
 Dark spots are voids; gray-shaded patches
 are second phase; ASTM grain size No. 11
 86-pct of theoretical density.



Sample No. B-942 Mag.: 500 X
 Ident. T-178-20 Etch: Murakamis
 Remarks: As-sintered 4 hrs at 2,000° C.
 Gray-outlined patches are second phase;
 ASTM grain size No. 9-10; 90-pct of
 theoretical density.



Sample No. B-943 Mag.: 500 X
 Ident. T-178-22 Etch: Murakamis
 Remarks: As-sintered 4 hrs at 2,200° C.
 Dark spots are voids; ASTM grain size
 No. 8-9; 94-5-pct of theoretical density.



Sample No. B-944 Mag.: 500 X
 Ident. T-178-N Etch: Murakamis
 Remarks: As-sintered 4 hrs at 2,400° C.
 Dark spots are voids; ASTM grain size
 No. 8; 95-pct of theoretical density.

FIGURE 6. - Photomicrographs of Blended Tungsten-25 Rhenium Mixtures of Elemental
 1-1/4 Micron Tungsten and 2.2 Micron Rhenium Sintered at Various
 Temperatures in Hydrogen

TABLE 3. - Specific surface area, particle size, and distribution for tungsten-25 rhenium

| | Elemental mixtures | | | |
|--|------------------------|--------------------------------|----------------------------------|--------------------------------|
| | Prealloyed | | Prepressed | |
| | Curtiss-Wright | Union Carbide ORPA-1 ORPA-2 | 1-1/4 micron W, 2.2 micron Re | 4.2 micron W, 2.2 micron Re |
| Average particle size (Fisher subsieve), microns | 1.17 | 0.6 0.7 | 1.4 | 3.4 |
| Specific surface area (B. E. T.), m ² /g | 0.678 | 1.03 0.76 | 0.168 | 0.145 |
| Particle size distribution: | Percent particle count | | | |
| Microns | | | | |

| | | | | |
|-------|------|------|------|------|
| 20-44 | 6.4 | | 5.0 | 6.0 |
| 15-20 | 7.0 | 10.8 | 5.5 | 2.2 |
| 10-15 | 2.5 | | 2.1 | 3.1 |
| 9-10 | 1.9 | | 3.0 | 0.6 |
| 8-9 | 1.7 | | 3.2 | 1.1 |
| 7-8 | 1.5 | 5.5 | 1.3 | 1.4 |
| 6-7 | 2.6 | 6.0 | 2.1 | 4.5 |
| 5-6 | 5.5 | 5.1 | 0.7 | 10.3 |
| 4-5 | 8.6 | | 3.6 | 19.4 |
| 3-4 | 8.1 | | 6.6 | 23.3 |
| 2-3 | 16.4 | | 11.8 | 19.5 |
| 1-2 | 15.9 | 6.0 | 23.6 | 5.4 |
| 0-1 | 21.8 | 82.9 | 32.2 | 3.2 |

TABLE 4. - X-ray diffraction and microprobe analyses of as-received prealloyed tungsten-25 rhenium powders

| | Average Re content, wt-pct | X-ray diffraction | | | Microprobe | |
|----------------|----------------------------|---------------------|--------------------------------|-------------------------------|---------------|-------------------------------------|
| | | Primary constituent | <u>1/</u> Lattice parameter, A | Minor consti. | Trace consti. | Re content, wt-pct extreme majority |
| Curtiss-Wright | 24. | W-Re solid solution | 3.1548 <u>2/</u> 3.1618 | Chi phase | Re | 5-83 8-10.5 |
| ORPA-1 | 24.2 | W-Re Solid solution | 3.1489 | Sigma phase | Re, chi phase | 12-42 18-28 |
| ORPA-2 | 26.6 | W-Re solid solution | 3.1489 | Sigma phase, W ₃ O | - | 10-54 21-54 |

1/ Primary = Up to 100%; Minor = 5 to 30%; Trace = less than 10%.

2/ Lattice parameters of: Tungsten = 3.1650 ± 0.0005 A
W-25Re = 3.1465 ± 0.0005 A

TABLE 5. - Data for prealloyed tungsten-25 rhenium evaluation sleeves sintered at various temperatures in hydrogen

| 1/ Sleeve number | Sintering treatment | Density | | Rhenium content, wt-pct | Grain size, ASTM no. | Second phase search | | | |
|------------------|---------------------|---------|------------------------|-------------------------|----------------------|---------------------|------------|-------------------|-----------------|
| | | g/cc | Percent of theoretical | | | Metallography | Microprobe | X-ray diffraction | Lattice para. A |
| CW-15 | 3/ | 17.65 | 89.9 | 24.3 | <12 | 10-15% | Some | trace | 3.151 |
| T-150-18 | 4/ | 17.51 | 89.3 | 23.7 | 10-11 | Some | trace | trace | 3.1475 |
| T-150-20 | 5/ | 18.15 | 92.6 | 23.6 | 9 | Some | None | None | 3.1477 |
| CW-20 | 5/ | 17.88 | 91.4 | 24.1 | 8 | None | <1% | trace | 3.1467 |
| T-150-22 | 6/ | 18.57 | 94.6 | 23.8 | 8 | 1-glob | 1-glob | None | 3.1477 |
| CW-N | 7/ | 18.73 | 95.4 | 24.3 | 6 | None | None | - | - |
| CW-4N | 8/ | 19.26 | 98.1 | 23.8 | 4 | None | - | - | - |
| T-152-15 | 3/ | 18.14 | 92.4 | 24.2 | <13 | 10-15% | - | trace | 3.1468 |
| -18 | 4/ | 18.28 | 93.1 | 24.2 | 11 | <5% | - | trace | 3.1479 |
| -20 | 5/ | 18.43 | 94.0 | 24.6 | 10 | 1-glob | None | None | 3.1474 |
| -22 | 6/ | 9/17.62 | 89.8 | 24.3 | 9 | None | None | None | 3.1482 |
| -N | 7/ | 18.25 | 93.1 | 24.4 | 7 | None | - | None | - |
| -4N | 8/ | 17.61 | 89.8 | 24.1 | 6 | None | - | None | 3.149 |
| T-180-15 | 3/ | 18.03 | 91.09 | 26.5 | <12 | 10% | - | trace | 3.1464 |
| -18 | 4/ | 19.05 | 97.1 | 26.7 | 9 | Some | 5% | None | 3.1456 |
| -20 | 5/ | 19.11 | 97.5 | 26.7 | 8 | Some | - | None | 3.1457 |
| -22 | 6/ | 19.37 | 98.6 | 26.8 | 5 | Some | <1% | None | 3.1460 |
| -N | 7/ | 19.52 | 99.5 | 26.8 | 5 | None | None | None | - |
| -4N | 8/ | 19.36 | 98.6 | 26.3 | 3 | None | - | None | 3.1456 |

(Continued)

TABLE 5. - Data for prealloyed tungsten-25 rhenium evaluation sleeves sintered at various temperatures in hydrogen (Continued)

| 1/ Sleeve number | Sintering treatment | Density | | Rhenium content, wt-pct | Grain size, ASTM no. | Second phase search | | |
|------------------|---------------------|---------|---------------------------|-------------------------|----------------------|---------------------|---------------|-----------------------------------|
| | | g/cc | 2/ Percent of theoretical | | | ASTM no. | Metallography | X-ray diffraction Lattice para. A |
| T-178-15 | 3/ | 9/ 16.1 | 82.1 | 25.3 | <12 | | Much | Much |
| -18 | 4/ | 16.86 | 85.9 | - | 11 | | 30% | <10% |
| -20 | 5/ | 9/ 17.7 | 90.2 | - | 10 | | 15-20% | <1% |
| -22 | 6/ | 18.51 | 94.4 | - | 9 | | None | None |
| -N | 7/ | 18.65 | 95.1 | - | 8 | | None | None |
| -4N | 8/ | 18.79 | 95.8 | 25.4 | 7 | | None | None |

- 1/ CW and T-150 = Curtiss-Wright material
T-152 = Union Carbide Lot #1 (ORPA-1)
T-180 = Union Carbide Lot #2 (ORPA-2)
- 2/ Based upon theoretical density of 19.625 g/cc.
- 3/ 4 hours at 1,500° C.
- 4/ 4 hours at 1,500° C plus 4 hours at 1,800° C.
- 5/ 4 hours at 1,500° C plus 4 hours at 2,000° C.
- 6/ 4 hours at 1,500° C plus 4 hours at 2,200° C.
- 7/ 4 hours at 1,500° C plus 4 hours at 2,400° C.
- 8/ 15 hours at 2,400° C.
- 9/ Porous sample.
- T-178 Not prealloyed, blended mixture elemental 1-1/4 micron tungsten and 2.2 micron rhenium.

degrees and (2) specimens free of second phase could be obtained by sintering at temperatures as low as $2,000^{\circ}\text{C}$, as compared to a sintering temperature of $2,200^{\circ} - 2,400^{\circ}\text{C}$ required for blended-elemental mixtures. The handling of prealloyed powders did not pose any problem and small evaluation size sleeves were processed without difficulty to yield sintered sleeves of a quality comparable to those concurrently made by the use of blended mixtures of elemental tungsten and rhenium.

RHENIUM AND RHENIUM-BASE ALLOYS

H. Kato, Project Coordinator

R. R. Lowery

This project was authorized to obtain engineering properties on the W-25 Re alloy. Alloys produced by both arc melting and powder metallurgy processes were to be investigated.

Powdered metal was the starting material for both processes. Chase Brass and Copper Company furnished the necessary 15 pounds of rhenium as 325 mesh grade I powder. Tungsten powder of 4.5 micron particle size was purchased from a commercial supplier. The powders were blended to the tungsten-25 weight-percent rhenium composition by screening and tumbling cycles.

Approximately 12 kilograms of alloy were prepared and isostatically pressed at 30,000 psi to form a compact for sintering. After a 2,450° C sinter for 15 hours in a hydrogen atmosphere, a powder metallurgy compact was obtained that finished to a 7.6 cm diameter by 11.5 cm long extrusion billet.

Some 14 kilograms of alloy were pressed into compacts suitable for consumable electrodes in the arc-melting cycle. These compacts also were isostatically pressed at 30,000 psi, then sintered in hydrogen for 8 hours at 1,500° C, and then for 2 hours at 2,100° C in vacuum. The arc melting of these electrodes produced a rough ingot 8.4 cm in diameter and 14.6 cm in height. This ingot was machined to an extrusion billet 7.6 cm in diameter by 12 cm long.

The two extrusion billets were sent to ORNL in early February, where the A. E. C. had made arrangements for them to be extruded to sheet bar. Late in April, word was received from Oak Ridge that difficulty had been encountered, consequently both the powder compact and the arc-melted ingot were not extruded successfully.

The billets were heated to 3,990° F and glass coated in both cases. The powder compact extrusion was attempted at a reduction ratio of 8 to 1 with an extrusion speed of 2.5 to 3 inches per second; the arc ingot was tried with a ratio of 6 to 1 and a speed of 5 inches per second. Molybdenum nose and follower plugs were used in both cases as were coated dies. Only one-half inch of the powder compact was extruded, with a slightly better yield of 4 inches for the arc-melted ingot. The 700-ton capacity extrusion press stalled at these points in both cases.

An attempt is being made by ORNL personnel to recover the remainder of the billets for another extrusion attempt.